SOLVAY
SODA ASH JOINT VENTURE

July 11, 1997

Bernie Dailey
WDEQ-Air Quality Division
122 W. 25th St.
Cheyenne, WY 82002

RE: Solvay Soda Ash Joint Venture Air Quality Permit Application AP-W77

Dear Bernie:

Following is an explanation of the VISCREEN inputs and the procedure for Methods 5 and 202.

VISCREEN Model Inputs:

The inputs for the VISCREEN model include all permitted particulate emission rates and the Method 202 back half organic emission rates. The permitted particulate emission rates include the Method 5 front half and Method 202 inorganic back half emissions. Through stack testing, it has been found that the only significant Method 202 back half organic emissions are in the calciner stack exhaust, therefore, only sources AQD #s 17, 48, and 82 have included Method 202 back half organic emissions for the VISCREEN model. These rates are noted in the table below:

EPA Method 202 Back Half Organic

AQD # - Unit	PPH	TPY
17 - Calciners #s 1 & 2 common stack	32.1	141
48 - Calciner #3	9.2	40
82 - Calciner #4	29.4	129
Total	70.7	310

NOTE: AQD #s 17 and 48 Method 202 organic emission rates are based on actual testing, the AQD #82 (proposed Calciner #4) emission rate was calculated based on testing of the existing calciners.

The total permitted particulate emissions following the proposed expansion will be 113 PPH or 495 TPY. The 310 TPY of Method 202 back half organic plus the 495 TPY of particulate emissions total 805 TPY of emissions for the VISCREEN model. The model was run with an input of 812 TPY of particulate. At the time of the model run, the estimated emissions were higher than the current estimate.

Methods 5 and 202:

The purpose of Method 5 is to determine particulate emissions from a stationary source. A sample of the stack exhaust is drawn through a heated probe and is passed through a 0.3 μm filter. The sample is then condensed in a series of impingers so that percent moisture of the stack exhaust can be determined.

Method 202 is a modification to the impingers or "back half" catch of the conventional Method 5 train. The purpose of this method is to quantify particulate matter that condenses after the Method 5 "front half" filter. The impingers are held at a temperature below 20°C (68°F). After the sampling is complete, the impinger catch is mixed with methylene chloride to extract the organic fraction. This fraction and the remaining water fraction are separated and allowed to evaporate to dryness, leaving behind a residue. Method 202 does not necessarily simulate normal post stack ambient conditions, and therefore, may not accurately determine that portion of the stack exhaust that condenses to particulate in the atmosphere. Solvay Soda Ash Joint Venture in concert with Tg Soda Ash, OCI Wyoming, and Clean Air Engineering are pursuing a rigorous analysis of the back half inorganic catch to determine if Method 202 is indeed applicable to the exhaust stream from a trona calciner. However, at this time, the permitted particulate emission rates include this inorganic portion of the Method 202 back half. Furthermore, per your request, the results of Method 202 back half organic have been added to the permitted particulate emission rates for use in the VISCREEN visibility model.

Enclosed you will find printouts of Methods 5 and 202, and a summary of each.

Sincerely,

Dolly A. Potter
Environmental Engineer

Enclosures

cc: Lee Gribovicz - WDEQ-AQD/Lander
   Tamara Blett - USFS/Lakewood
   Ann Mebane - USFS/Pinedale

METHOD 5 — DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1.1   Principle
   * gas sample drawn isokinetically from gas stream
   * filter maintained at a prescribed temperature
   * particulate matter collected onto a glass fiber filter
   * particulate mass determined gravimetrically, after removal of water

1.2   Applicability
   * determination of particulate emissions from stationary sources

2.1   Sampling Train
   * sampling train as shown in Figure 5-1
   * construction details given in APTD-0581
   * operation and maintenance described in APTD-0576

2.1.1   Probe Nozzle
   * stainless steel, glass or approved
   * button-hook or elbow
   * sharp leading edge with < 30° outside taper
   * 1/8 to 1/2 in diameter (0.3 to 1.2 cm) or larger
   * each nozzle marked and calibrated

2.1.2   Probe Liner
   * borosilicate or quartz glass (or metal if approved)
   * equipped with probe heater (to ~120°C or as specified)
   * borosilicate liners OK up to 480°C (900°F)
     quartz liners OK up to 900°C (1650°F)
   * 316 stainless steel and incoloy 825 OK, subject to approval

2.1.3   Pitot Tube
   * type S, attached to probe
   * pitot impact tube and sampling nozzle as shown in Figure 2-6B
   * pitot calibrated as detailed in Method 2 (pitot tube shall have a known coefficient)

2.1.4   Differential Pressure Gauge
   * inclined manometer or equivalent
   * one for pitot and one for flow orifice

2.1.5   Filter Holder
   * borosilicate glass with glass frit and silicon gasket
   * other filter holders OK if approved
   * holder must seal (pass leak test)

2.1.6   Filter Heating System
   * required to keep glass filter dry
   * capable of 120°C or specified temperature
   * equipped with ± 3°C temperature measurement

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2.1.7   Condenser
    * four impingers in series, leak-free fittings
    * second impinger of Greenburg-Smith design
    * modifications are subject to approval
    * known water in impinger one and two
    * known weight of silica gel, or equivalent, in impinger four
    * equipped with outlet temperature $\pm$ 1°C
    * condenser outlet temperature of < 20°C
    * other condenser systems subject to approval

2.1.8   Metering System
    * equipment as shown in Figure 5-1
    * leak-free pump, with vacuum gauge
    * dry gas meter $\pm$ 2%, with thermometer $\pm$ 3°C
    * flow rate meter

2.1.9   Barometer
    * capable of measuring $\pm$ 2.5 mm Hg (0.1 inch)
    * or corrected reading from weather station
    * adjusted to sampling height (1" Hg per 1000 feet)

2.1.10   Gas Density Determination Equipment
    * refer to Methods 2, 3 and 4

2.2   Sample Recovery
    * probe-liner and probe-nozzle nylon brushes
    * wash bottles, glass or polyethylene
    * sample storage containers, glass or polyethylene
    * petri dishes, for filter
    * graduated cylinder or balance
    * plastic storage container for silica gel
    * funnel

2.3   Analysis
    * glass weighing dishes
    * desiccator
    * analytical balance, $\pm$ 0.1 mg
    * balance $\pm$ 0.5 g
    * beakers, etc.

3.1   Sampling

3.1.1   Filters
    * glass fiber without organic binder
    * 99.95% efficient at 0.3 μm
    * unreactive to SO_2 or SO_3

3.1.2   Silica Gel
    * 6 to 16 mesh
    * indicating type
    * redry at 175°C for 2 hours

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3.2   Sample Recovery
* acetone-reagent grade, <0.001% residue

4.1   Sampling Procedure
* tester should be trained and experienced
* weigh several 200-300 g portions of silica gel (or weight silica gel added to impinger)
* check, label, condition and weight filter
* condition filter > 24 hr in desiccator or > 2 hr in oven (105°C)
* select sampling site and points by Method 1
* determine velocity pressure by Method 2
* determine gas density by Methods 3 and 4
* measure stack and port, clean out port opening
* select nozzle, probe and probe liner
* mark distances on sampling probe
* determine sampling time requirements
* prepare and assemble sampling train (including water and silica gel in impingers, filter in filter holder and ice and water in condenser)
* leak-check system, if desired, at >15" Hg vacuum
* release vacuum from nozzle end
* post-test leak-check required
* operate train at isokinetic rate (± 10%) at each sampling point by adjusting flow rate
* sample equal time at each point
* leak-check during run if components changed
* read, adjust and maintain proper filter box temperature
* heat and adjust probe temperature to avoid condensation
* level and zero manometers before test
* record initial gas meter valve
* position nozzle at first sampling position
* start sampling at pre-determined sampling rate
* read velocity pressure and adjust flow to isokinetic rate
* record all temperatures (probe, hot box, impinger and gas meter)
* record velocity pressure, orifice pressure and vacuum
* at end of sampling point time move probe to next position
* maintain sampling while moving probe
* record approximate gas meter volume at time of moving
* adjust sampling rate to new isokinetic value
* adjust probe and hot box temperature as needed
* add ice to condenser, if needed
* record all temperatures and pressure valves
* if adjustments are necessary, record values and times
* after sampling at final point, close coarse flow valve and turn off pump
* withdraw probe from stack
* if stack is at high negative pressure (vacuum), start/stop sampling pump with probe outside stack
* record final gas meter volume
* conduct post-test leak-check

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* leak rate less than 4% or 0.02 cfm acceptable, otherwise correction required (or void test)
* separate (break) connection between filter outlet and condenser inlet
* recover sample, analyze and calculate results

4.2   Sample recovery
* at end of sampling, probe is removed from stack, leak check conducted and probe allowed to cool
* remove probe from sampling train, clean off ends (silicone grease) and cap end
* remove umbilical cord
* remove and drain flexible filter-impinger connection line (if used)
* clean and cap off impinger (condenser section) inlet and outlet
* remove, clean and cap filter assembly
* transfer probe, filter assembly and condenser to cleanup area (this area should be clean and protected from the wind)
* inspect sampling train components and record any abnormal conditions, such as a bent nozzle or unusual condenser water color.
* remove filter from filter holder and place in its marked petri dish (or similar container)
* if necessary, clean any remaining filter fibers from holder or gasket and add to filter container
* quantitatively recover particulate matter from probe nozzle, fittings and liner plus that from all glassware at inlet side of filter
* clean above parts by washing/rinsing with acetone and brushing with Nylon bristle brush (wash/rinse with water if required)
* brush/rinse parts until no visible particle remains
* brush glass probe three times or more
* brush metal probe six times or more
* store cleaning catch in container #2
* mark liquid level in container and identify
* save sample of acetone (or water) for a blank analysis
* determine water condensate in impingers by weight ($\pm$ 0.5g) or by volume measurement ($\pm$ 1 ml)
* determine weight change of silica gel (note color of indicating silica gel)
* save liquid impinger catch only if an analysis is required. Save in marked container
* record weight/volume gain on data sheet.

4.3   Sample analysis
Container No. 1 - Filter
* desiccate 24+ hours to "constant weight" (weight change of <0.5 mg or 1% of gain)
* or oven dry at 105°C for 2+ hours
* weigh to $\pm$ 0.1 mg

Container No. 2 - probe wash
* check liquid level (for correction if necessary)
* transfer liquid wash to a tared beaker (250 ml)
* evaporate to dryness
* desiccate to constant weight
* weigh residue to $\pm$ 0.1 mg

Container No. 3 - silica gel
* weight to $\pm$ 0.5 mg (may be done in field)

Container with Acetone (or water) Blank
* measure liquid sample by volume or by weight
* transfer to tared beaker
* evaporate and desiccate
* weigh to $\pm$ 0.1 mg

4.4   Quality Control Procedures
* check meter orifice value by calculation
* check gas meter Y factor
* use calibrated orifice to check flow

5.   Calibration Procedures

5.1   Probe Nozzle
* measure and mark before initial use
* using micrometer to measure inside diameter $\pm$ 0.001"
* average measurements in three directions
* individual measurement differences $\leq$ 0.004"
* record values and nozzle No. in log
* nozzle inlet edge to be sharp and round
* if diameter difference > 0.004" reshape and remeasure

5.2   Pitot Tube
* check pitot tube dimensions as in Method 2

5.3   Metering System
* calibrate system prior to field use and after field use
* calibrate system against reference test meter (wet or dry) of $\pm$ 1% accuracy
* use equipment set up as shown in Figure 5.5
* conduct a metering system leak-check
* conduct initial calibrations at several flow rates (3 minimum)
* record data and calculations as shown in Figure 5.6
* check allowable tolerance of Y and H_a values
* recalibrate system after each field use
* conduct three tests at one intermediate flow rate
* calibrate at maximum system vacuum reached during field test
* if Y factor changes more than 5%, recalibrate at all flow rates

5.4   Probe Heater Calibration
       * check probe heating system before initial use
       * refer to APTD-0576 for calibration procedure
       * refer to APTD-0581 for probe construction details
       * probes with outlet temperature monitors need not be calibrated

5.5   Temperature Gauges
       * calibrate meter system temperature gauges by comparison with
         calibrated lab thermometer.
       * calibrate stack gas temperature by Method 2 procedure

5.6   Leak Check of Metering System
       * set up system as shown in Figure 5-4
       * pressurize to about 15cm water and observe manometer for one
         minute
       * a loss of pressure indicates a leak
       * correct leak and retest

6.   Calculations
       * calculate average gas meter temperature
       * calculate average orifice pressure drop
       * calculate (or measure) pressure at sampling port
       * calculate dry gas volume sampled
       * calculate (if necessary) leak rate correction
       * calculate water vapor volume
       * calculate moisture content
       * calculate acetone (or water) blank correction
       * calculate filter weight gain
       * calculate probe wash residue
       * calculate total particulate concentration
       * calculate percent isokinetic (I)
       * check acceptable %I (90%<I<110%)
       * calculate corrected concentration (for %I error)
       * calculate stack gas velocity
       * calculate volumetric flow rate

7.   Alternative Procedures

7.1   Dry Gas Meter as a Calibration Standard
       * requires initial and periodic calibration
       * calibrate against spirometer or wet test meter
       * test set up shown in Figure 5-7
       * example data sheet shown in Figure 5-8
       * make triplicate runs at 5 flow rates from 10 to 34 lpm
       * calculate and compare "Y" factor for each test run
       * each flow Y factor range within 3%
       * meter coefficient between 0.95 and 1.05
       * plot a Q vs. average Y factor calibration curve
       * use this curve when calibrating Method 5 meter
       * recalibrate the standard dry gas meter annually or every 200
         hours of operation
       * recalibration may be performed at only two flow rates

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7.2   Critical Orifices as Calibration Standards
       * may be used in place of wet test meter
       * select five orifices to cover the 10 to 34 lpm flow range
         at least three orifices must be used to calibrate a Method 5
         dry gas meter
       * needle size recommendations are presented in a table
       * a convenient adapter is shown in Figure 5-9
       * use calibration apparatus setup as shown in Figure 5-10
       * conduct duplicate runs, calculate K using Equation 5-9
       * calibration procedure outline in Section 7.2.2.2
       * compare Y factor from two orifices, recalibrate if >2% change

40 CFR Part 40 Appendix A

EPA Method 5

Particulate Emissions

Air Quality Planning and Standards, Research Triangle Park, NC. Publication No. APTD-0581. 1973.

2. Devorkin, Howard, et al. Air Pollution Source Testing Manual. Air Pollution Control District, Los Angeles, CA. November, 1963.

3. Methods for Determination of Velocity, Volume, Dust, and Mist Content of Gases. Western Precipitation Division of Joy Manufacturing Co., Los Angeles, CA. Bulletin WP-50. 1968.

METHOD 5—DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1. Principle and Applicability

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature in the range of 120±14°C (248±25°F) or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator, for a particular application. The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method is applicable for the determination of particulate emissions from stationary sources.

2. Apparatus

2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 5-1. Complete construction details are given in APTD-0581 (Citation 2 in Bibliography); commercial models of this train are also available. For changes from APTD-0581 and for allowable modifications of the train shown in Figure 5-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576 (Citation 3 in Bibliography). Since correct usage is important in obtaining valid results, all users should read APTD-0576 and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. The sampling train consists of the following components:

ing the condenser, each to within 1 ml or 1 g may be used, subject to the approval of the Administrator. Acceptable means are to measure the condensed water either gravimetrically or volumetrically and to measure the moisture leaving the condenser by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures; or (2) passing the sample gas stream through a tared silica gel (or equivalent desiccant) trap with exit gases kept below 20° C (68° F) and determining the weight gain.

If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

NOTE II a determination of the particulate matter collected in the impingers is desired in addition to moisture content, the impinger system described above shall be used, without modification. Individual States or control agencies requiring this information shall be contacted as to the sample recovery and analysis of the impinger contents.

2.1.8 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 5-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APDT-0576 may be used provided that the specifications of this method are met.

2.1.9 Barometer. Mercury aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases the barometric reading may be obtained from a nearby National Weather Service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.10 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3. The temperature sensor shall, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see Method 2, Figure 2-7). As a second alternative, if a difference of not more than 1 percent in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube. (This alternative is subject to the approval of the Administrator.)

2.2 Sample Recovery. The following items are needed.

2.2.1 Probe-Liner and Probe-Nozzle Brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush shall have extensions (at least as long as the probe) of stainless steel, Nylon, Teflon, or similarly inert material. The brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

2.2.2 Wash Bottles—Two. Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

2.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for acetone washes, 500 ml or 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by acetone. (Narrow mouth glass bottles have been found to be less prone to leakage.) Alternatively, polyethylene bottles may be used.

2.2.4 Petri Dishes. For filter samples, glass or polyethylene, unless otherwise specified by the Administrator.

2.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances is suitable or use here and in Section 2.3.4.

2.2.6 Plastic Storage Containers. Air-tight containers to store silica gel.

2.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container, not necessary if silica gel is weighed in the field.

2.2.8 Funnel. Glass or polyethylene, to aid in sample recovery.

2.1.1 Probe Nozzle. Stainless steel (316) or glass with sharp, tapered leading edge. The angle of taper shall be ≤30° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing; other materials of construction may be used, subject to the approval of the Administrator.

A range of nozzle sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (⅛ to ½ in.)—or larger if higher volume sampling trains are used—inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in Section 5.

2.1.2 Probe Liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of 120±14° C (248±25° F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. (The tester may opt to operate the equipment at a temperature lower than that specified.) Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) will be considered acceptable.

Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480° C (900° F); quartz liners shall be used for temperatures between 480 and 900° C (900 and 1,650° F). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate is 820°C (1,508°F), and for quartz it is 1,500° C (2,732° F).

Whenever practical, every effort should be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners (e.g. 316 stainless steel, Incoloy 825, or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator.

2.1.3 Pitot Tube. Type S, as described in Section 2.1 of Method 2, or other device approved by the Administrator. The pitot tube shall be attached to the probe (as shown in Figure 5-1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of Method 2.

2.1.4 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.2 of Method 2. One manometer shall be used or velocity head (Δp) readings, and the other, for orifice differential pressure readings.

2.1.5 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other materials of construction (e.g. stainless steel, Teflon, Viton) may be used, subject to approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe (or cyclone, if used).

2.1.6 Filter Heating System. Any heating system capable of maintaining a temperature around the filter holder during sampling of 120±14° C (248±25° F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. Alternatively, the tester may opt to operate the equipment at a temperature lower than that specified. A temperature gauge capable of measuring temperature to within 3° C (5.4° F) shall be installed so that the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

2.1.7 Condenser. The following system shall be used to determine the stack gas moisture content. Four impingers connected in series with leak-free ground glass fittings or any similar leak-free non-contaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm (½ in.) ID glass tube extending to about 1.3 cm (½ in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Modifications (e.g. using flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator. The first and second impingers shall contain known quantities of water (Section 4.1.3), the third shall be empty, and the fourth shall contain a known weight of silica gel, or equivalent desiccant. A thermometer, capable of measuring temperature to within 1° C (2° F) shall be placed at the outlet of the fourth impinger for monitoring purposes.

Alternatively, any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving

2.3 Analysis. For analysis, the following equipment is needed.

2.3.1 Glass Weighing Dishes.

2.3.2 Desiccator.

2.3.3 Analytical Balance. To measure within 0.1 mg.

2.3.4 Balance. To measure to within 0.5 g.

2.3.5 Beakers, 250 ml.

2.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.

2.3.7 Temperature Gauge. To measure the temperature of the laboratory environment.

3. Reagents

3.1 Sampling. The reagents used in sampling are as follows:

3.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3-micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Standard Method D2986–71 (Reapproved 1978) (incorporated by reference—see § 60.17). Test data from the supplier's quality control program are sufficient for this purpose. In sources containing SO₂ or SO₃, the filter material must be of a type that is unreactive to SO₂ or SO₃. Citation 10 in Bibliography may be used to select the appropriate filter.

3.1.2 Silica Gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.3 Water. When analysis of the material caught in the impingers is required, deionized distilled water shall be used. Run blanks prior to field use to eliminate a high blank on test samples.

3.1.4 Crushed Ice.

3.1.5 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Administrator.

3.2 Sample Recovery. Acetone-reagent grade, ≤0.001 percent residue, in glass bottles—is required. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers; thus, acetone blanks shall be run prior to field use and only acetone with low blank values (≤0.001 percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

3.3 Analysis. Two reagents are required for the analysis:

3.3.1 Acetone. Same as 3.2.

3.3.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Administrator.

4. Procedure

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. It is suggested that sampling equipment be maintained according to the procedure described in APTD-0576.

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in the impinger or sampling holder just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label filters of the proper diameter on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass or plastic petri dishes) and keep the filters in these containers at all times except during sampling and weighing.

Desiccate the filters at 20±5.6° C (68±10° F) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e., 0.5 mg change from previous weighing; record results to the nearest 0.1 mg. During each weighing the filter must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent. Alternatively (unless otherwise specified by the Administrator), the filters may be oven dried at 105° C (220° F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Administrator.

4.1.2 Preliminary Determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 3.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for

the same total length of time as, the particulate sample run.

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of Method 2).

Select a suitable probe liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that (1) the sampling time per point is not less than 2 min (or some greater time interval as specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors. The sampling time at each point shall be the same.

In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

When glass liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260° C

(500° F) and an asbestos string gasket when temperatures are higher. See APTD-0576 for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Set up the train as in Figure 5-1, using (if necessary) a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by silicone grease. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack gas.

Place crushed ice around the impingers.

4.1.4 Pretest Leak-Check Procedure. A pretest leak-check is recommended, but not required. If the tester opts to conduct the pretest leak-check, the following procedure shall be used.

After the sampling train has been assembled, turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum.

NOTE.—A lower vacuum may be used, provided that it is not exceeded during the test.

If an asbestos string is used, do not connect the probe to the train during the leak-check. Instead, leak-check the train by first plugging the inlet to the filter holder (cyclone, if applicable) and pulling a 380 mm Hg (15 in. Hg) vacuum (see Note immediately above). Then connect the probe to the train and leak-check at about 25 mm Hg (1 in. Hg) vacuum; alternatively, the probe may be leak-checked with the rest of the sampling train, in one step, at 380 mm Hg (15 in. Hg) vacuum. Leakage rates in excess of 4 percent of the average sampling rate or 0.00057 m³/min (0.02 cfm), whichever is less, are unacceptable.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with bypass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse direction of bypass valve; this will cause water

FIGURE 5-2—PARTICULATE FIELD DATA

Plant _____
Location _____
Operator _____
Date _____
Run No _____
Sample box No _____
Meter box No _____
Meter ΔH@ _____
C factor _____
Pitot tube coefficient Cp _____

Ambient temperature _____
Barometric pressure _____
Assumed moisture, % _____
Probe length, m (ft.) _____
Nozzle identification No. _____
Average calibrated nozzle diameter, cm (in.) _____
Probe heater setting _____
Leak rate, m³/min. (cfm) _____
Probe liner material _____
Static pressure, mm Hg (in. Hg) _____
Filter No. _____

SCHEMATIC OF STACK CROSS SECTION

Traverse point number	Sampling time (θ), min.	Vacuum mm Hg (in. Hg)	Stack temperature (T_s), °C (°F)	Velocity head (ΔP_s), mm H_2O	Pressure differential across orifice meter (ΔH) mm H_2O (in. H_2O)	Gas sample volume m³ (ft³)	Gas sample temperature at dry gas meter Inlet °C (°F)	Gas sample temperature at dry gas meter Outlet °C (°F)	Filter holder temperature °C (°F)	Temperature of gas leaving condenser or last impinger °C (°F)
Total										
Average			Avg.	Avg.			Avg.	Avg.		

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o back up into the filter holder. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak-check as shown below and start over.

When the leak-check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable) and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and silica gel from being entrained backward into the third impinger.

4.1.4.2 Leak-Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak-check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure outlined in Section 4.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

Immediately after component changes, leak-checks are optional; if such leak-checks are done, the procedure outlined in Section 4.1.4.1 above shall be used.

4.1.4.3 Post-test Leak-Check. A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 4.1.4.1, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume

as shown in Section 6.3 of this method, or shall void the sampling run.

4.1.5 Particulate Train Operation. During the sampling run, maintain an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator) and a temperature around the filter of 120±14° C (248±25° F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator.

For each run, record the data required on a data sheet such as the one shown in Figure 5-2. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak-check, and when sampling is halted. Take other readings required by Figure 5-2 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

Clean the portholes prior to the test run to minimize the chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify that the filter and probe heating systems are up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient is 0.85±0.02, and the stack gas equivalent density (dry molecular weight) is equal to 29±4. APTD-0576 details the procedure for using the nomographs. If C_p and M_d are outside the above stated ranges do not use the nomographs unless appropriate steps (see Citation 7 in Bibliography) are taken to compensate for the deviations.

When the stack... consider significant nega-
tive pressure (height of impinger stem,
take care to close the coarse adjust valve
before inserting the probe into the stack to
prevent water from backing into the filter
holder. If necessary, the pump may be
turned on with the coarse adjust valve
closed.

When the probe is in position, block off
the openings around the probe and porthole
to prevent unrepresentative dilution of the
gas stream.

Traverse the stack cross-section, as re-
quired by Method 1 or as specified by the
Administrator, being careful not to bump
the probe nozzle into the stack walls when
sampling near the walls or when removing
or inserting the probe through the port-
holes; this minimizes the chance of extract-
ing deposited material.

During the test run, make periodic adjust-
ments to keep the temperature around the
filter holder at the proper level; add more
ice and, if necessary, salt to maintain a tem-
perature of less than 20° C (68° F) at the
condenser/silica gel outlet. Also, periodical-
ly check the level and zero of the manome-
ter.

If the pressure drop across the filter be-
comes too high, making isokinetic sampling
difficult to maintain, the filter may be re-
placed in the midst of a sample run. It is
recommended that another complete filter
assembly be used rather than attempting to
change the filter itself. Before a new filter
assembly is installed, conduct a leak-check
(see Section 4.1.4.2). The total particulate
weight shall include the summation of all
filter assembly catches.

A single train shall be used for the entire
sample run, except in cases where simulta-
neous sampling is required in two or more
separate ducts or at two or more different
locations within the same duct, or, in cases
where equipment failure necessitates a
change of trains. In all other situations, the
use of two or more trains will be subject to
the approval of the Administrator.

Note that when two or more trains are
used, separate analyses of the front-half
and (if applicable) impinger catches from
each train shall be performed, unless identi-
cal nozzle sizes were used on all trains, in
which case, the front-half catches from the
individual trains may be combined (as may
the impinger catches) and one analysis of
front-half catch and one analysis of imping-
er catch may be performed. Consult with
the Administrator for details concerning the
calculation of results when two or more
trains are used.

At the end of the sample run, turn off the
coarse adjust valve, remove the probe and
nozzle from the stack, turn off the pump,
record the final dry gas meter reading, and
conduct a post-test leak-check, as outlined
in Section 4.1.4.3. Also, leak-check the pilot

lines as described in Method 2, Section 3.1;
the lines must pass this leak-check, in order
to validate the velocity head data.

4.1.6 Calculation of Percent Isokinetic.
Calculate percent isokinetic (see Calcula-
tions, Section 6) to determine whether the
run was valid or another test run should be
made. If there was difficulty in maintaining
isokinetic rates due to source conditions,
consult with the Administrator for possible
variance on the isokinetic rates.

4.2 Sample Recovery. Proper cleanup
procedure begins as soon as the probe is re-
moved from the stack at the end of the sam-
pling period. Allow the probe to cool.

When the probe can be safely handled,
wipe off all external particulate matter near
the tip of the probe nozzle and place a cap
over it to prevent losing or gaining particu-
late matter. Do not cap off the probe tip
tightly while the sampling train is cooling
down as this would create a vacuum in the
filter holder, thus drawing water from the
impingers into the filter holder.

Before moving the sample train to the
cleanup site, remove the probe from the
sample train, wipe off the silicone grease,
and cap the open outlet of the probe. Be
careful not to lose any condensate that
might be present. Wipe off the silicone
grease from the filter inlet where the probe
was fastened and cap it. Remove the umbili-
cal cord from the last impinger and cap the
impinger. If a flexible line is used between
the first impinger or condenser and the
filter holder, disconnect the line at the
filter holder and let any condensed water or
liquid drain into the impingers or condens-
er. After wiping off the silicone grease, cap
off the filter holder outlet and impinger
inlet. Either ground-glass stoppers, plastic
caps, or serum caps may be used to close
these openings.

Transfer the probe and filter-impinger as-
sembly to the cleanup area. This area
should be clean and protected from the
wind so that the chances of contaminating
or losing the sample will be minimized.

Save a portion of the acetone used for
cleanup as a blank. Take 200 ml of this ace-
tone directly from the wash bottle being
used and place it in a glass sample container
labeled "acetone blank."

Inspect the train prior to and during dis-
assembly and note any abnormal conditions.
Treat the samples as follows:

Container No. 1. Carefully remove the
filter from the filter holder and place it in
its identified petri dish container. Use a pair
of tweezers and/or clean disposable surgical
gloves to handle the filter. If it is necessary
to fold the filter, do so such that the partic-
ulate cake is inside the fold. Carefully trans-
fer to the petri dish any particulate matter
and/or filter fibers which adhere to the
filter holder gasket, by using a dry Nylon

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brush and/or a sharp-edged blade.
Seal the container.

Container No. 2. Taking care to see that
dust on the outside of the probe or other ex-
terior surfaces does not get into the sample,
quantitatively recover particulate matter or
any condensate from the probe nozzle,
probe fitting, probe liner, and front half of
the filter holder by washing these compo-
nents with acetone and placing the wash in
a glass container. Distilled water may be
used instead of acetone when approved by
the Administrator and shall be used when
specified by the Administrator. In these
cases, save a water blank and follow the Ad-
ministrator's directions on analysis. Perform
the acetone rinses as follows:

Carefully remove the probe nozzle and
clean the inside surface by rinsing with ace-
tone from a wash bottle and brushing with
a Nylon bristle brush. Brush until the ace-
tone rinse shows no visible particles, after
which make a final rinse of the inside sur-
face with acetone.

Brush and rinse the inside parts of the
Swagelok fitting with acetone in a similar
way until no visible particles remain.

Rinse the probe liner with acetone by tilt-
ing and rotating the probe while squirting
acetone into its upper end so that all inside
surfaces will be wetted with acetone. Let the
acetone drain from the lower end into the
sample container. A funnel (glass or poly-
ethylene) may be used to aid on transfer-
ring liquid washes to the container. Follow
the acetone rinse with a probe brush. Hold
the probe in an inclined position, squirt ace-
tone into the upper end as the probe brush
is being pushed with a twisting action
through the probe; hold a sample container
underneath the lower end of the probe, and
catch any acetone and particulate matter
which is brushed from the probe. Run the
brush through the probe three times or
more until no visible particulate matter is
carried out with the acetone or until none
remains in the probe liner on visual inspec-
tion. With stainless steel or other metal
probes, run the brush through the above
prescribed manner at least six times since
metal probes have small crevices in which
particulate matter can be entrapped. Rinse
the brush with acetone, and quantitatively
collect these washings in the sample con-
tainer. After the brushing, make a final ace-
tone rinse of the probe as described above.

It is recommended that two people clean
the probe to minimize sample losses. Be-
tween sampling runs, keep brushes clean
and protected from contamination.

After ensuring that all joints have been
wiped clean of silicone grease, clean the
inside of the front half of the filter holder
by rubbing the surfaces with a Nylon bristle
brush and rinsing with acetone. Rinse each
surface three times or more if needed to
remove visible particulate. Make a final

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rinse of the brush and... (if appli-
cable). After all acetone washings and par-
ticulate matter have been collected in the
sample container, tighten the lid on the
sample container so that acetone will not
leak out when it is shipped to the labora-
tory. Mark the height of the fluid level to
determine whether or not leakage occurred
during transport. Label the container to
clearly identify its contents.

Container No. 3. Note the color of the
indicating silica gel to determine if it has
completely spent and make a notation of its
condition. Transfer the silica gel from the
fourth impinger to its original container
and seal. A funnel may make it easier to
pour the silica gel without spilling. A rubber
policeman may be used as an aid in remov-
ing the silica gel from the impinger. It is not
necessary to remove the small amount of
dust particles that may adhere to the im-
pinger wall and are difficult to remove.
Since the gain in weight is to be used for
moisture calculations, do not use any water
or other liquids to transfer the silica gel. If
a balance is available in the field, follow the
procedure for container No. 3 in Section 3.3.

Impinger Water. Treat the impingers as
follows: Make a notation of any color or
film in the liquid catch. Measure the liquid
which is in the first three impingers to
within ±1 ml by using a graduated cylinder
or by weighing it to within ±0.5 g by using a
balance (if one is available). Record the
volume or weight of liquid present. This in-
formation is required to calculate the mois-
ture content of the effluent gas.

Discard the liquid after measuring and re-
cording the volume or weight, unless analy-
sis of the impinger catch is required (see
Note, Section 2.1.7).

If a different type of condenser is used,
measure the amount of moisture condensed
either volumetrically or gravimetrically.

Whenever possible, containers should be
shipped in such a way that they remain up-
right at all times.

4.3 Analysis. Record the data required on
a sheet such as the one shown in Figure 5-3.
Handle each sample container as follows:

Plant —————
Date —————
Run No. —————
Filter No. —————
Amount liquid lost during transport —————
Acetone blank volume, ml —————
Acetone wash volume, ml —————
Acetone blank concentration, mg/mg (Equa-
tion 5-4) —————
Acetone wash blank, mg (Equation 5-5) —————

FIGURE 5-3—ANALYTICAL DATA

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stant weight. Report the results to the nearest 0.1 mg.

Container No. 3. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. This step may be conducted in the field.

"Acetone Blank" Container. Measure acetone in this container either volumetrically or gravimetrically. Transfer the acetone to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

NOTE: At the option of the tester, the contents of Container No. 2 as well as the acetone blank container may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature must be below the boiling point of the solvent; also, to prevent "bumping," the evaporation process must be closely supervised, and the contents of the beaker must be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

4.4 Quality Control Procedures. The following quality control procedures are suggested to check the volume metering system calibration values at the field test site prior to sample collection. These procedures are optional for the tester.

4.4.1 Meter Orifice Check. Using the calibration data obtained during the calibration procedure described in Section 5.3, determine the $\Delta H@$ for the metering system orifice. The $\Delta H@$ is the orifice pressure differential in units of in. H_2O that correlates to 0.75 cfm of air at 528°R and 29.92 in. Hg. The $\Delta H@$ is calculated as follows:

$$\Delta H@ = 0.0319 \, \Delta H \left[\frac{T_m}{P_{bar}} \frac{\theta'}{Y^2 V_m} \right] \quad \text{Eq. 5-9}$$

Where:
ΔH=Average pressure differential across the orifice meter, in. H_2O.
T_m=Absolute average dry gas meter temperature, °R.
P_{bar}=Barometric pressure, in. Hg.
θ=Total sampling time, min.
Y=Dry gas meter calibration factor, dimensionless.
V_m=Volume of gas sample as measured by dry gas meter, dcf.
$0.0319=(0.0567 \text{ in. Hg/°R}) \times (0.75 \text{ cfm})^2$.

Container number	Weight of particulate collected, mg		
	Final weight	Tare weight	Weight gain
1.			
2.			
Total			
Less acetone blank			
Weight of particulate meter			

$$\frac{\text{Increase, g}}{(1 \text{ g/ml})} = \text{Volume water, ml}$$

"Convert weight of water to volume by dividing total weight increase by density of water (1 g/ml).

	Volume of liquid water collected	
	Impinger volume, ml	Silica gel weight, g
Final		
Initial		
Liquid collected		
Total volume collected		

_____ θ' _____ ml

Container No. 1. Leave the contents in the shipping container or transfer the filter and any loose particulate from the sample container to a tared glass weighing dish. Desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg. For purposes of this Section, the term "constant weight" means a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings.

Alternatively, the sample may be oven dried at 105° C (220° F) for 2 to 3 hours, cooled in the desiccator, and weighed to a constant weight, unless otherwise specified by the Administrator. The tester may also oven dry the sample at 105° C (220° F) for 2 to 3 hours, weigh the sample, and use this weight as a final weight.

Container No. 2. Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a con-

Before beginning the field test (a set of three runs usually constitutes a field test), operate the metering system (i.e., pump, volume meter, and orifice) at the $\Delta H@$ pressure differential for 10 minutes. Record the volume collected, the dry gas meter temperature, and the barometric pressure. Calculate a dry gas meter calibration check value, Y_c, as follows:

$$Y_c = \frac{10}{V_m} \left[\frac{0.0319 \, T_m}{P_{bar}} \right]^{\frac{1}{2}} \quad \text{Eq. 5-10}$$

Where:
Y_c=Dry gas meter calibration check value, dimensionless.
10=10 minutes of run time.

Compare the Y_c value with the dry gas meter calibration factor Y to determine that:

$$0.97Y < Y_c < 1.03Y$$

If the Y_c value is not within this range, the volume metering system should be investigated before beginning the test.

4.4.2 Calibrated Critical Orifice. A calibrated critical orifice, calibrated against a wet test meter or spirometer and designed to be inserted at the inlet of the sampling meter box may be used as a quality control check by following the procedure of Section 7.2.

5. Calibration

Maintain a laboratory log of all calibrations.

5.1 Probe Nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated

before use. Each nozzle shall be permanently and uniquely identified.

5.2 Pitot Tube. The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 4 of Method 2.

5.3 Metering System.

5.3.1 Calibration Prior to Use. Before its initial use in the field, the metering system shall be calibrated as follows: Connect the metering system inlet to the outlet of a wet test meter that is accurate to within 1 percent. Refer to Figure 5.5. The wet test meter should have a capacity of 30 liters/rev (1 ft³/rev). A spirometer of 400 liters (14 ft³) or more capacity, or equivalent, may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should be periodically calibrated with a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes may be used, provided that the specified accuracies of the procedure are maintained. Run the metering system pump for about 15 minutes with the orifice manometer indicating a median reading as expected in field use to allow the pump to warm up and to permit the interior surface of the wet test meter to be thoroughly wetted. Then, at each of a minimum of three orifice manometer settings, pass an exact quantity of gas through the wet test meter and note the gas volume indicated by the dry gas meter. Also note the barometric pressure, and the temperatures of the wet test meter, the inlet of the dry gas meter, and the outlet of the dry gas meter. Select the highest and lowest orifice settings to bracket the expected field operating range of the orifice. Use a minimum volume of 0.15 m³ (5 cf) at all orifice settings. Record all the data on a form similar to Figure 5.6, and calculate Y, the dry gas meter calibration factor, and $\Delta H@$, the orifice calibration factor, at each orifice setting as shown on Figure 5.6. Allowable tolerances for individual Y and $\Delta H@$ values are given in Figure 5.6. Use the average of the Y values in the calculations in Section 6.

METERING SYSTEM   WET TEST METER

THERMOMETER

U-TUBE MANOMETER

Figure 5.5   Equipment arrangement for metering system calibration.

Date _____

Barometric pressure, P_b = _____ in. Hg

Metering System Identification: _____

Orifice manometer (wet meter) setting ΔH in. H_2O	Spirometer (wet meter) gas volume V_w ft.3	Dry gas meter volume V_m ft.3	Temperatures				
			Spirometer (wet meter) t_w °F	Dry Gas Meter			
				Inlet t_i °F	Outlet t_o °F	Average t_m °F	Time θ min

Calculations

ΔH in. H_2O	Y	$\Delta H@$
	$\dfrac{V_w \, P_b \, (t_m + 460)}{V_m \left[P_b + \dfrac{\Delta H}{13.6} \right] (t_w + 460)}$	$\dfrac{0.0317 \, \Delta H}{P_b \, (t_o + 460)} \left[\dfrac{(t_w + 460) \, \theta}{V_w} \right]^2$

Average _____

Y   = Ratio of reading of wet test meter to dry test meter; tolerance for individual values $\pm$0.02 from average.

$\Delta H@$ = Orifice pressure differential that equates to 0.75 cfm of air @ 68°F and 29.92 inches of mercury, in. H_2O; tolerance for individual values $\pm$0.20 from average.

Figure 5.6.   Example data sheet for calibration of metering system (English units).

Figure 5-4. Leak check of meter box.

Before calibrating the metering system, it is suggested that a leak-check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: make a 10-minute calibration run at 0.00057m³/min (0.02 cfm); at the end of the run, take the difference of the measured wet test meter and dry gas meter volumes; divide the difference by 10, to get the leak rate. The leak rate should not exceed 0.00057 m³/min (0.02 cfm).

5.3.2 Calibration After Use. After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single, intermediate orifice setting (based on the previous field test), with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate the average value of the dry gas meter calibration factor. If the value has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as previously detailed.

Alternative procedures, e.g., rechecking the orifice meter coefficient may be used, subject to the approval of the Administrator.

5.3.3 Acceptable Variation in Calibration. If the dry gas meter coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

5.4 Probe Heater Calibration. The probe heating system shall be calibrated before its initial use in the field. Use a heat source to generate air heated to selected temperatures that approximate those expected to occur in the sources to be sampled. Pass this air through the probe at a typical simple flow rate while measuring the probe inlet and outlet temperatures at various probe heater settings. For each air temperature generated, construct a graph of probe heating system setting versus probe outlet temperature. The procedure outlined in APTD-0576 can also be used. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used. Also, probes with outlet temperature monitoring capabilities do not require calibration.

5.5 Temperature Gauges. Use the procedure in Section 4.3 of Method 2 to calibrate in-stack temperature gauges. Dial thermometers, such as are used for the dry gas meter and condenser outlet, shall be calibrated against mercury-in-glass thermometers.

5.6 Leak Check of Metering System Shown in Figure 5-1. That portion of the sampling train from the pump to the orifice meter should be leak checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 5-4): Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for one minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

5.7 Barometer. Calibrate against a mercury barometer.

6. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.
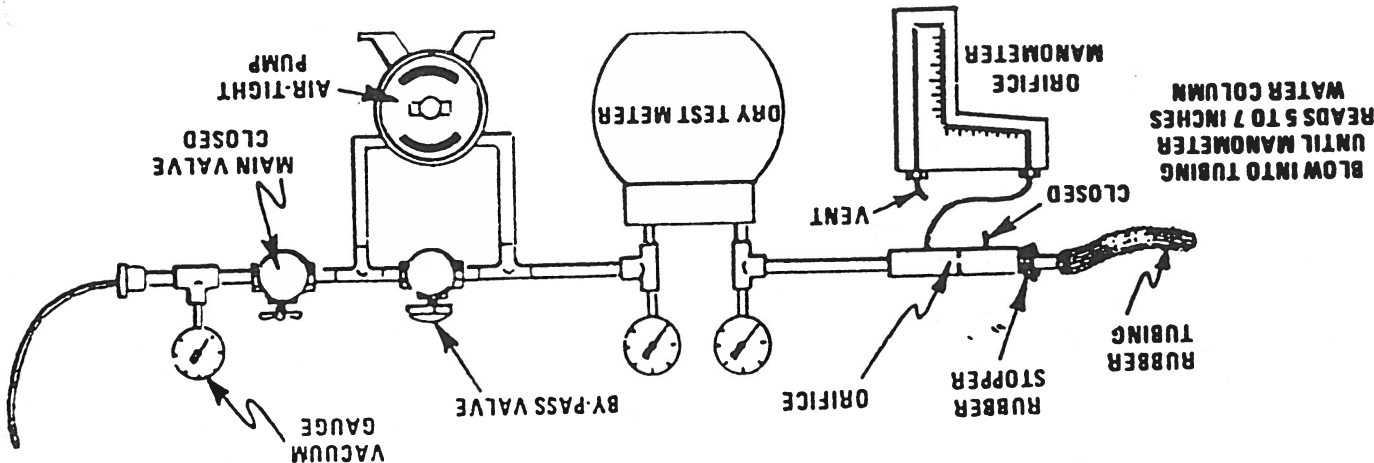
6.1 Nomenclature.

A_n = Cross-sectional area of nozzle, m^2 (ft^2).

B_{ws} = Water vapor in the gas stream, proportion by volume.

C_a = Acetone blank residue concentration, mg/mg.

c_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).

I = Percent of isokinetic sampling.

L_a = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to 0.00057 m^3/min (0.02 cfm) or 4 percent of the average sampling rate, whichever is less.

L_i = Individual leakage rate observed during the leak check conducted prior to the "ith" component change ($i = 1, 2, 3...n$), m^3/min (cfm).

L_p = Leakage rate observed during the post-test leak check, m^3/min (cfm).

m_a = Mass of residue of acetone after evaporation, mg.

m_n = Total amount of particulate matter collected, mg.

M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).

P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).

P_s = Absolute stack gas pressure, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 mm Hg-m^3/°K-g-mole (21.85 in. Hg-ft^3/°R-lb-mole).

T_m = Absolute average dry gas meter temperature (see Figure 5-2), °K (°R).

T_s = Absolute average stack gas temperature (see Figure 5-2), °K (°R).

T_{std} = Standard absolute temperature, 293°K (528°R).

V_a = Volume of acetone blank, ml.

V_{aw} = Volume of acetone used in wash, ml.

V_{lc} = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.

V_m = Volume of gas sample as measured by dry gas meter, dcm (dscf).

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).

$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).

v_s = Stack gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 5, m/sec (ft/sec).

W_a = Weight of residue in acetone wash, mg.

Y = Dry gas meter calibration factor.

ΔH = Average pressure differential across the orifice meter (see Figure 5-2), mm H_2O (in. H_2O).

ρ_a = Density of acetone, mg/ml (see label on bottle).

ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).

θ = Total sampling time, min.

θ_1 = Sampling time interval, from the beginning of a run until the first component change, min.

θ_i = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.

θ_n = Sampling time interval, from the final (nth) component change until the end of the sampling run, min.

13.6 = Specific gravity of mercury.

60 = Sec/min.

100 = Conversion to percent.

6.2 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop. See data sheet (Figure 5-2).

6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20°C, 760 mm Hg or 68°F, 29.92 in. Hg) by using Equation 5-1.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right) \left[\frac{P_{bar} + (\Delta H/13.6)}{P_{std}}\right]$$

$$= K_1 V_m Y \frac{P_{bar} + (\Delta H/13.0)}{T_m}$$

Equation 5-1

Where:

K_1 = 0.3858 °K/mm Hg for metric units

= 17.64 °R/in. Hg for English units

NOTE: Equation 5-1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., the post-test leak check or leak checks conducted prior to component changes) exceeds L_a. If L_p or i exceeds L_a, Equation 5-1 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace V_m in Equation 5-1 with the expression:

$$[V_m - (L_p - L_a)\theta]$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace V_m in Equation 5-1 by the expression:

$$V_m - (L_1 - L_a)\theta_1$$

$$-\sum_{i=2}^{n} (L_i - L_a)\theta_i - (L_p - L_a)\theta_p$$

and substitute only for those leakage rates (L_i or L_p) which exceed L_a.

6.4 Volume of Water Vapor.

$$V_{w(std)} = \frac{V_{lc} \rho_w R T_{std}}{M_w P_{std}} = K_2 V_{lc}$$

Eq. 5-2

Where:

K_2 = 0.001333 m^3/ml for metric units

= 0.04707 ft^3/ml for English units.

6.5 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}}$$

Eq. 5-3

NOTE: In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger analysis (Equation 6-3), and a second from the assumption of saturated conditions. The lower of the two values of B_{ws} shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in the Note of Section 1.2 of Method 4. For the purposes of this method, the average stack gas temperature from Figure 5-2 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is ±1°C (2°F).

6.6 Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_a \rho_a}$$

Eq. 5-4

6.7 Acetone Wash Blank.

$$W_a = C_a V_{aw} \rho_a$$

Eq. 5-5

6.8 Total Particulate Weight. Determine the total particulate catch from the sum of the weights obtained from Containers 1 and 2 less the acetone blank (see Figure 5-3).

NOTE: Refer to Section 4.1.5 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

6.9 Particulate Concentration.

$$c_s = (0.001 \, g/mg)(m_n/V_{m(std)})$$

Eq. 5-6

6.10 Conversion Factors:

From	To	Multiply by
scf	m^3	0.02832
g/ft^3	mg/m^3	0.001
	g/ft^3	15.43
	g/m^3	2.205 × 10⁻³
	g/ft^3	35.31

6.11 Isokinetic Variation.

6.11.1 Calculation From Raw Data.

$$I = \frac{100 T_s [K_3 V_{lc} + (V_m Y/T_m)(P_{bar} + \Delta H/13.6)]}{60 \theta v_s P_s A_n}$$

Eq. 5-7

Where:

K_3 = 0.003454 mm Hg-m^3/ml-°K for metric units.

= 0.002669 in. Hg-ft^3/ml-°R for English units.

6.11.2 Calculation From Intermediate Values.

$$I = \frac{100 T_s V_{m(std)} P_{std}}{T_{std} v_s \theta A_n P_s 60 (1 - B_{ws})}$$

$$= \frac{K_4 T_s V_{m(std)}}{P_s v_s A_n \theta (1 - B_{ws})}$$

Eq. 5-8

Where:

K_4 = 4.320 for metric units

= 0.09450 for English units.

6.12 Acceptable Results. If 90 percent ≤ I ≤ 110 percent, the results are acceptable. If the particulate results are low in comparison to the standard, and if I is over 110 percent or less than 90 percent, the Administrator may opt to accept the results. Citation 4 in the bibliography section can be used to make acceptability judgments. If I is judged to be unacceptable, reject the particulate results and repeat the test.

6.13 Stack Gas Velocity and Volumetric Flow Rate. Calculate the average stack gas velocity and volumetric flow rate, if needed, using data obtained in this method and equations in Sections 6.2 and 6.3 of Method 2.

7.1 Alternative Procedures

7.1 Dry Gas Meter as a Calibration Standard. A dry gas meter may be used as a calibration standard for volume measurements in place of the wet test meter specified in Section 8.3, provided that it is calibrated initially and recalibrated periodically as follows:

7.1.1 Standard Dry Gas Meter Calibration.

$$Q = 17.65 \cdot \frac{V_s}{\theta} \cdot \frac{P_b}{(t_s + 460)}$$

$$Y_{ds} = \frac{V_s}{V_{ds}} \cdot \frac{(t_{ds} + 460)}{(t_s + 460)} \cdot \frac{P_b}{P_b + \frac{\Delta p}{13.6}}$$

Figure 5.8. Example data sheet for calibration of a standard dry gas meter for method 5 sampling equipment (English units).

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DATE: _____

DRY GAS METER IDENTIFICATION: _____

BAROMETRIC PRESSURE (P_b): _____ in. Hg

APPROXIMATE FLOW RATE (Q) cfm	DRY GAS VOLUME (WET METER) (V_s) ft³	DRY GAS METER VOLUME (WET METER) (V_{ds}) ft³	SPIROMETER (WET METER) (t_s) °F	TEMPERATURES DRY GAS METER			DRY GAS METER PRESSURE (Δp) in. H₂O	TIME (θ) min.	FLOW RATE (Q) cfm	METER COEFFICIENT (Y_{ds})	AVERAGE METER COEFFICIENT (Y_{ds})
				INLET (t_i) °F	OUTLET (t_o) °F	AVERAGE (t_d) °F					
0.40											
0.60											
0.80											
1.00											
1.20											

7.1.1.1 The dry gas meter to be calibrated and used as a secondary reference meter should be of high quality and have an appropriately sized capacity, e.g., 3 liters/rev (0.1 ft³/rev). A spirometer (400 liters or more capacity), or equivalent, may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should have a capacity of 30 liters/rev (1 ft³/rev) and capable of measuring volume to within ±1.0 percent; wet test meters should be checked against a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes may be used, provided that the speci-

fied accuracies of the procedure are maintained.

7.1.1.2 Set up the components as shown in Figure 5.7. A spirometer, or equivalent, may be used in place of the wet test meter in the system. Run the pump for at least 5 minutes at a flow rate of about 10 liters/min (0.35 cfm) to condition the interior surface of the wet test meter. The pressure drop indicated by the manometer at the inlet side of the dry gas meter should be minimized [no greater than 100 mm H₂O (4 in. H₂O) at a flow rate of 30 liters/min (1 cfm)]. This can be accomplished by using large diameter tubing connections and straight pipe fittings.
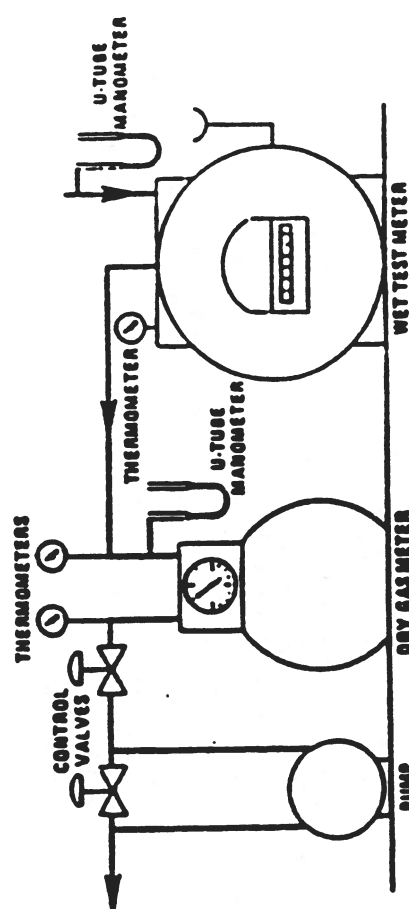
Figure 5.7 . Equipment arrangement for dry-gas meter calibration.

7.1.1.3 Collect the data as shown in the example data sheet (see Figure 5-8). Make triplicate runs at each of the flow rates and at no less than five different flow rates. The range of flow rates should be between 10 and 34 liters/min (0.35 and 1.2 cfm) or over the expected operating range. The

7.1.1.4 Calculate wet test meter gas volume, V_w, and the run time, θ. Calculate the dry gas meter coefficient, Y_{ds}, for each run. These calculations are as follows:

$$Y_{ds} = \frac{V_w\,(t_{ds} + t_w)}{V_s\,(t_s + t_w)} \cdot \frac{P_{bar}}{P_{bar} + \dfrac{\Delta p}{13.6}}$$

$$Q = K_1\,\frac{P_{bar}}{t_s + t_w}\,\frac{V_s}{\theta}$$

Where:

K_1 = 0.3858 for International system of units (SI): 17.64 for English units.
V_w = Wet test meter volume, liters (ft³).
V_s = Dry gas meter volume, liters (ft³).
t_{ds} = Average dry gas meter temperature, °C (°F).
t_w = 273° C for SI units; 460° F for English units.
t_s = Average wet test meter temperature, °C (°F).
θ = Run time, min.
P_{bar} = Barometric pressure, mm Hg (in. Hg).
Δp = Dry gas meter inlet differential pressure, mm H₂O (in. H₂O).

7.1.1.5 Compare the three Y_{ds} values at each of the flow rates and determine the maximum and minimum values. The difference between the maximum and minimum values at each flow rate should be no greater than 0.030. Extra sets of triplicate runs may be made in order to complete this requirement. In addition, the meter coefficients should be between 0.95 and 1.05. If these specifications cannot be met in three sets of successive triplicate runs, the meter is not suitable as a calibration standard and should not be used as such. If these specifications are met, average the three Y_{ds} values at each flow rate resulting in five average meter coefficients, Y_{ds}.

7.1.1.6 Prepare a curve of meter coefficient, Y_{ds}, versus flow rate, Q, for the dry gas meter. This curve shall be used as a reference when the meter is used to calibrate other dry gas meters and to determine whether recalibration is required.

7.1.2 Standard Dry Gas Meter Recalibration.

7.1.2.1 Recalibrate the standard dry gas meter against a wet test meter or spirometer annually or after every 200 hours of operation, whichever comes first. This requirement is valid provided the standard dry gas meter is kept in a laboratory and, if transported, cared for as any other laboratory instrument. Abuse to the standard meter may cause a change in the calibration and will require more frequent recalibrations.

7.1.2.2 As an alternative to full recalibration, a two-point calibration check may be made. Follow the same procedure and equipment arrangement as for a full recalibration, but run the meter at only two flow rates [suggested rates are 14 and 28 liters/min (0.5 and 1.0 cfm)]. Calculate the meter coefficients for these two points, and compare the values with the meter calibration curve. If the two coefficients are within ±1.5 percent of the calibration curve values at the same flow rate, the meter need not be recalibrated until the next date for a recalibration check.

7.2 Critical Orifices As Calibration Standards. Critical orifices may be used as calibration standards in place of the wet test meter specified in Section 5.3, provided that they are selected, calibrated, and used as follows:

7.2.1 Section of Critical Orifices.

7.2.1.1 The procedure that follows describes the use of hypodermic needles or stainless steel needle tubings which have been found suitable for use as critical orifices. Other materials and critical orifice designs may be used provided the orifices act as true critical orifices; i.e., a critical vacuum can be obtained, as described in Section 7.2.2.3. Select five critical orifices that are appropriately sized to cover the range of flow rates between 10 and 34 liters/min or the expected operating range. Two of the critical orifices should bracket the expected operating range.

A minimum of three critical orifices will be needed to calibrate a Method 5 dry gas meter (DGM): the other two critical orifices can serve as spares and provide better selection for bracketing the range of operating flow rates. The needle sizes and tubing lengths shown below give the following approximate flow rates:

Gauge/cm	Flow rate (liters/min)	Gauge/cm	Flow rate (liters/min)
12/7.8	32.56	14/2.5	19.54
12/10.2	30.02	14/5.1	17.27
13/2.5	25.77	14/7.6	16.14
13/5.1	23.50	15/3.2	14.16
13/7.6	22.37	15/7.6	11.81
13/10.2	20.67	15/10.2	10.48

7.2.1.2 These needles can be adapted to a Method 5 type sampling train as follows: Insert a serum bottle stopper, 13- by 20-mm sleeve type, into a ¾-inch Swagelok quick connect. Insert the needle into the stopper as shown in Figure 5-9.
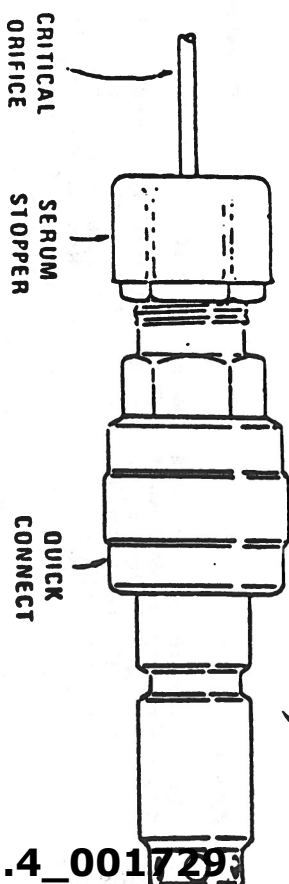
Figure 5-9. Critical orifice adaptation to Method 5 metering system.

CRITICAL ORIFICE
SERUM STOPPER
QUICK CONNECT

7.2.2 Critical Orifice Calibration. The procedure described in this section uses the Method 5 meter box configuration with a DGM as described in Section 2.1.8 to calibrate the critical orifices. Other schemes may be used, subject to the approval of the Administrator.

7.2.2.1 Calibration of Meter Box. The critical orifices must be calibrated in the same configuration as they will be used; i.e., there should be no connections to the inlet of the orifice.

7.2.2.1.1 Before calibrating the meter box, leak check the system as follows: Fully open the coarse adjust valve, and completely close the by-pass valve. Plug the inlet. Then turn on the pump, and determine whether there is any leakage. The leakage rate shall be zero; i.e., no detectable movement of the DGM dial shall be seen for 1 minute.

7.2.2.1.2 Check also for leakages in that portion of the sampling train between the pump and the orifice meter. See Section 8.0 for the procedure; make any corrections, if necessary. If leakage is detected, check for cracked gaskets, loose fittings, worn O-rings, etc., and make the necessary repairs.

7.2.2.1.3 After determining that the meter box is leakless, calibrate the meter box according to the procedure given in Section 5.3. Make sure that the wet test meter meets the requirements stated in Section 7.1.1.1. Check the water level in the wet test meter. Record the DGM calibration factor, Y.

7.2.2.2 Calibration of Critical Orifices. Set up the apparatus as shown in Figure 5-10.
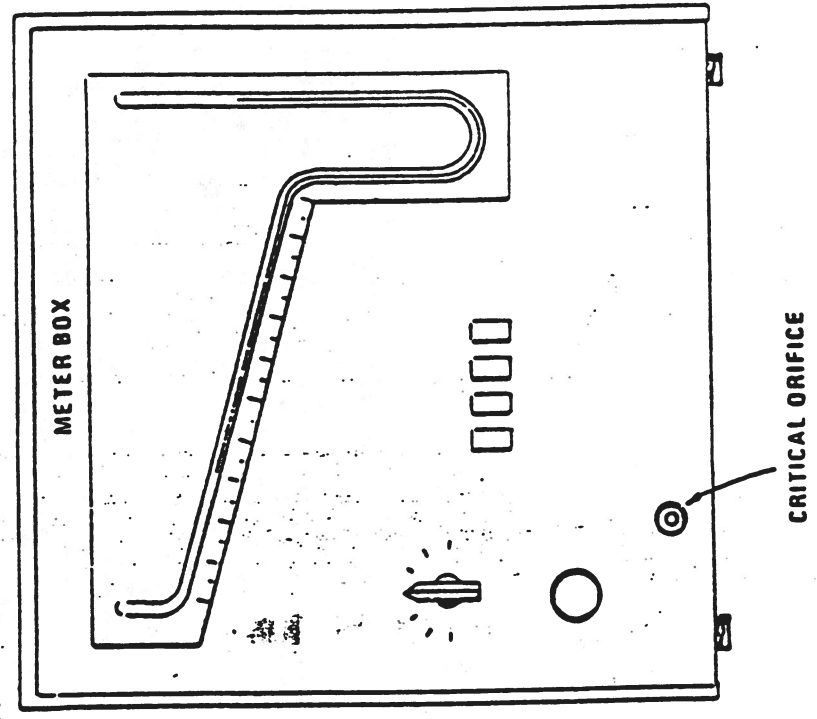
METER BOX

CRITICAL ORIFICE

Figure 5-10. Apparatus setup.

7.2.2.1 Allow a warm-up time of 15 minutes. This step is important to equilibrate the temperature conditions through the DGM.

7.2.2.2 Leak check the system as in Section 7.2.2.1.1. The leakage rate shall be zero.

7.2.3 Before calibrating the critical orifice, determine its suitability and the appropriate operating vacuum as follows: Turn on the pump, fully open the coarse adjust valve, and adjust the by-pass valve to give a vacuum reading corresponding to about half of atmospheric pressure. Observe the meter box orifice manometer reading, ΔH. Slowly increase the vacuum reading until a stable reading is obtained on the meter box orifice manometer. Record the critical vacuum for each orifice.

Orifices that do not reach a critical value shall not be used.

7.2.2.4 Obtain the barometric pressure using a barometer as described in Section 2.1.9. Record the barometric pressure, P$_{bar}$, in mm Hg (in. Hg).

7.2.2.5 Conduct duplicate runs at a vacuum of 25 to 50 mm Hg (1 to 2 in. Hg) above the critical vacuum. The runs shall be at least 5 minutes each. The DGM volume readings shall be in increments of 0.00283 m³ (0.1 ft³) or in increments of complete revolutions of the DGM. As a guideline, the times should not differ by more than 3.0 seconds (this includes allowance for changes in the DGM temperature) to achieve ± 0.5 percent in K'. Record the information listed in Figure 5-11.

7.2.2.6 Calculate K' using Equation 5-9.

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$$K' = \frac{K_1 V_m (P_{bar} + \Delta H/13.6)\sqrt{T_{amb}}}{P_{bar} T_m \theta}$$

Eq. 5-9

Where:

K' = Critical orifice coefficient,

$\sqrt{T_{amb}}$ = Absolute ambient temperature, °K (°R).

Average the K' values. The individual K' values should not differ by more than ±0.5 percent from the average.

7.2.3 Using the Critical Orifices as Calibration Standards.

7.2.3.1 Record the barometric pressure.

Date _____ Train ID _____ DGM cal.
factor _____ Critical orifice ID _____

Dry gas meter		Run number	
		1	2
Final reading	m' (ft³)	/	/
Initial reading	m' (ft³)	/	/
Difference, V$_m$	m' (ft³)	/	/
Inlet/Outlet temperatures:			
Initial	°C (°F)	/	/
Final	°C (°F)	/	/
Avg. Temperature, t$_m$	°C (°F)	/	/
Time, θ	min./sec.	/	/
	min		
Orifice man. rdg., ΔH	mm (in.) H$_2$O	/	/
Bar. pressure, P$_{bar}$	mm (in.) Hg		
Ambient temperature, t$_{amb}$	°C (°F)		
Pump vacuum	mm (in.) Hg		
K' factor			
Average			

Figure 5-11. Data sheet for determining K' factor.

7.2.3.2 Calibrate the metering system according to the procedure outlined in Sections 7.2.2.1 to 7.2.2.5. Record the information listed in Figure 5.12.

7.2.3.3 Calculate the standard volumes of air passed through the DGM and the critical orifices, and calculate the DGM calibration factor, Y, using the equations below:

$$V_{m(std)} = K_1 V_m \frac{(P_{bar} + \Delta H/13.6)}{T_m}$$

Eq. 5-10

$$V_{cr(std)} = K' V_m$$

Eq. 5-11

$$V_{cr(std)} = K' \sqrt{\frac{P_{bar} \theta}{T_{amb}}}$$

$$Y = \frac{V_{cr(std)}}{V_{m(std)}}$$

Eq. 5-12

where:

V$_{cr(std)}$ = Volume of gas sample passed through the critical orifice, corrected to standard conditions, dsm³ (dscf).

K$_1$ = 0.3858 °K/mm Hg for metric units = 17.64 °R/in. Hg for English units.

7.2.3.4 Average the DGM calibration values for each of the flow rates. The calibration factor, Y, at each of the flow rates should not differ by more than 2 percent from the average.

7.2.3.5 To determine the need for recalibrating the critical orifices, compare the DGM Y factors obtained from two adjacent orifices each time a DGM is calibrated; for example, when checking 13/2.5, use orifices 12/10.2 and 13/5.1. If any critical orifice yields a DGM Y factor differing by more than 2 percent from the others, recalibrate the critical orifice according to Section 7.2.2.

Date _____ Train ID _____ Critical ori-
fice ID _____ Critical orifice K' factor
_____

		Run number
		2
Dry gas meter		
Final reading	m' (ft³)	
Initial reading	m' (ft³)	

Dry gas meter		Run number	1	2
Difference, V_m:	m³ (ft³)		/	/
Inlet/outlet temperature:				
Initial	°C (°F)		/	/
Final	°C (°F)		/	/
Avg. Temperature, t_m	°C (°F)		/	/
Time, θ	min/sec.		/	/
Orifice man. rdg., ΔH	mm (in.) H₂O		/	/
Bar. pressure, P_{bar}	mm (in.) Hg		/	/
Ambient temperature, t_a	°C (°F)		/	/
Pump vacuum	mm (in.) Hg		/	/
$V_{m(std)}$	m³ (ft³)		/	/
DGM cal. factor, Y			/	/

Figure 5-12. Data sheet for determining DGM Y factor.

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2. Apparatus

2.1 Sampling Train. The sampling train configuration is the same as shown in Figure 5-1 of Method 5. The sampling train consists of the following components:

2.1.1 Probe Nozzle, Pitot Tube, Differential Pressure Gauge, Filter Holder, Condenser, Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 2.1.1, 2.1.3 to 2.1.5, and 2.1.7 to 2.1.10, respectively.

2.1.2 Probe Liner. Same as in Method 5, Section 2.1.2, with the note that at high stack gas temperatures (greater than 250°C (480°F)), water-cooled probes may be required to control the probe exit temperature to 42±10°C (108±18°F).

2.1.3 Precollector Cyclone. Borosilicate glass following the construction details shown in Air Pollution Technical Document-0581, "Construction Details of Isokinetic Source-Sampling Equipment".

NOTE: The tester shall use the cyclone when the stack gas moisture is greater than 10 percent. The tester shall not use the precollector cyclone under other, less severe conditions.

2.1.4 Filter Heating System. Any heating (or cooling) system capable of maintaining a sample gas temperature at the exit end of

METHOD 5A—DETERMINATION OF PARTICULATE EMISSIONS FROM THE ASPHALT PROCESSING AND ASPHALT ROOFING INDUSTRY

1. Applicability and Principle

1.1 Applicability. This method applies to the determination of particulate emissions from asphalt roofing industry process saturators, blowing stills, and other sources as specified in the regulations.

1.2 Principle. Particulate matter is withdrawn isokinetically from the source and collected on a glass filter maintained at a temperature of 42±10°C (108±18°F). The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water.

11. Westlin, P. R. and R. T. Shigehara. Procedure for Calibrating and Using Dry Gas Volume Meters as Calibration Standards. Source Evaluation Society Newsletter. 3(1):17-30. February 1978.

12. Lodge, J.P. Jr., J. B. Pate, B.E. Ammons, and G.A. Swanson. The Use of Hypodermic Needles as Critical Orifices in Air Sampling. J. Air Pollution Control Association. 16:197-200. 1968.

METHOD 6—DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

1. Principle and Applicability

1.1 Principle. A gas sample is extracted from the sampling point in the stack. The sulfuric acid mist (including sulfur trioxide) and the sulfur dioxide are separated. The sulfur dioxide fraction is measured by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfur dioxide emissions from stationary sources. The minimum detectable limit of the method has been determined to be 3.4 milligrams (mg) of SO₂/m³ ($2.12×10^{-7}$ lb/ft³). Although no upper limit has been established, tests have shown that concentrations as high as 80,000 mg/m³ of SO₂ can be collected efficiently in two midget impingers, each containing 15 milliliters of 3 percent hydrogen peroxide, at a rate of 1.0 lpm for 20 minutes. Based on theoretical calculations, the upper concentration limit in a 20-liter sample is about 93,300 mg/m³.

Possible interferents are free ammonia, water-soluble cations, and fluorides. The ammonia is removed by the glass wool filters and the isopropanol bubbler, and hence do not affect the SO₂ analysis. When samples are being taken from a gas stream with high concentrations of very fine metallic fumes (such as in inlets to control devices), a high-efficiency glass fiber filter must be used in place of the glass wool plug (i.e., the one in the probe) to remove the metallic fumes.

Free ammonia interferes by reacting with SO₂ to form particulate sulfite and by reacting with the indicator. If free ammonia is present (this can be determined by knowledge of the process and noticing white particulate matter in the probe and isopropanol bubbler), the alternative procedures in Section 7.2 shall be used.

METHOD 202 - DETERMINATION OF CONDENSIBLE PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1.1   Applicability
   * determination of condensible particulate matter (CPM) from stationary sources
   * defined as material that condenses after passing through an in-stack filter
   * method intended for use with Method 201 or 201A
   * in general, could be used with Methods 5 or 17
   * specifies use of glass lined probe
   * method could be modified for other probe or filter temperature
   * other absorbed gases could become part of the above defined CPM

1.2   Principle
   * CPM is collected in the impinger portion of a Method 17 train
   * impingers are purged with nitrogen to remove dissolved sulfur dioxide gases
   * impinger solution extracted with methylene chloride
   * organic and aqueous fractions taken to dryness and residue weighed
   * sum of two fractions represents CPM

2.1   Precision
   * a few select data indicates approximately ±20%

2.2   Interference
   * ammonia (i.e. as used to control HCl)
   * can measure Cl and correct CPM value

3.   Apparatus
   * same as Method 17 except
   * glass lined probe
   * first two impingers of standard tip design
   * pre cleaned and $MeCl_2$ rinsed glassware
   * sample recovery as in Method 5, plus
   * Nitrogen cylinder and purge line
   * with rotameter for 20 lpm N_2 flow rate
   * ion chromatograph used for sulfate analysis
   * sampling train shown in Figure 202-1
   * post-test nitrogen purge system shown in Figure 202-2

4.   Reagents
   * in general, reagents of Method 5
   * deionized distilled water
   * N_2 gas
   * methylene chloride
   * ammonium hydroxide
   * pH indicator (phenolphthalein)

5.   Procedure
   * in general, same as Method 5
   * place 100ml water in first three impingers

* silicone grease use is not recommended
* in general, sample recovery as in Method 17
* post-test N_2 purge recommended, but optional
* if little SO_2 present (impinger Ph > 4.5), purging not necessary
* detach probe and purge, with ice in impinger box (maintain gas temp. < 20°C)
* purge for 1 hour at 20 lpm
* operate meter box pump at < 20 lpm
* sample handling as follows:
* Container #1, #2 and #3 - as in Method 5, 201, etc.
* Container #4 - Impinger contents
    * measure liquid volume or weight
    * transfer to clean sample bottle
    * rinse twice with water
    * make sample bottle and liquid level
* Container #5 - $MeCl_2$ rinse
    * rinse twice with $MeCl_2$
    * save rinse in clean glass jar
    * mark bottle and level
* Container #6 - water blank
* Container #7 - $MeCl_2$ blank
* Container #8 - acetone blank

5.3   Analysis
* record data on sheet such as Figure 202-3
* Containers #1, #2 and #3 - as detailed in Method 5 or 201 etc.
* Container #4 and #5 - organic fraction
    * measure liquid volume of each container
    * save 5 ml aliqual of #4 for IC analysis of sulfates
    * extract organics by mixing #4 and #5 contents in a 1000ml separatory funnel
    * after separation, drain off $MeCl_2$ phase
    * add 75ml $MeCL_2$ to funnel, mix and drain
    * repeat with another 75ml $MeCl_2$
    * each time, leave some $MeCl_2$ in funnel to ensure no water collected in organic phase
    * place extract in tared 350ml weighing tin
    * evaporate at room temperature and pressure
    * desiccate 24 hr. and weigh
* Container #4 and #5 - inorganic fraction
    * evaporate aqueous phase to 50ml on a hot plate
    * redissolve residue in 100ml water
    * add fine drops phenolphthalein
    * add concentrated NH_4OH until sample turns pink
    * evaporate to dryness in a 105°C oven
    * desiccate 24 hr. and weigh
    * NH_4OH addition is optional when little SO_2 present i.e. if pH > 4.5, NH_4OH not necessary
* Container #4 aliquot - sulfate
    * refer to Method 57 for IC Sulfate analysis
    * if NH_4OH not added omit this step
    * determine sulfate in a aliquot by IC
    * calculate correction factor

.6   Barometer
          * calibrate against mercury-in-glass barometer

5.7   Probe cyclone and nozzle
          * need not be calibrated if cyclone meets Figure 12
            specifications and nozzle meet appendix B specifications
          * if not, test cyclone and nozzle combination against
            specifications of Table 1
          * perform triplicate test at the 15 conditions of Table 2
          * perform tests in a suitable wind tunnel
          * calibration requires generation and measurement of solid,
            monodispersed test aerosol
          * from the calibration data, determine the cyclone flow rate at
            which D_{50} is 10μm
          * for acceptance of cyclone, data must fall within limits of
            Figure 13
          * cyclone flow rate, gas viscosity, gas density and D_{50}
            relationship must be determined
          * calibration must cover general range of interest or use

6.   Calculations
          * the EGR data reduction computer program can be used to make
            the necessary calculations. Example inputs and outputs are
            shown in Figure 14
          * calculations can be done manually, as specified in Method 5,
            with equations given in method

6.2 PM_{10} particulate weight
          * sum of container #1 and #3 (less acetone blank)

6.3   Total particulate weight
          * sum of containers #1, #2 and #3 (less acetone blank)

6.4 PM_{10} fraction
          * PM_{10} divided by total

6.5   Cyclone flow rate
          * at standard conditions, use equation on page 14253
          * at actual conditions, use equation on page 14254

6.6   Aerodynamic cut size
          * calculate water fraction (in stack gas)
          * calculate gas viscosity (in cyclone)
          * calculate molecular weight (of stack gas)
          * calculate cyclone actual D_{50}

6.7   Acceptable results
          * 9μm < D_{50} < 11μm
          * 90% < I < 110%
          * if D_{50} > 11μm, results may be accepted
          * if D_{50} < 9μm, results rejected

* Container #6 and #7
    * analyze blanks as for Container #4 and #5 above
* Container #8
    * analyze acetone blank as in Method 5

6.   Calibration
* follow calibrations of Method 5
* Calibrate IC as in Method 57

7.   Calculations
* follow calculations of Method 5
* correct for NH_4^+ and H_2O
* use equation 202-1 for NH_4 added
* calculate inorganic CPM by equation 202-2
* calculate CPM concentration by equation 202-3

8.   Alternative Procedures
* determine NH_4 by titration
* analyze chlorides by IC
* use air purge to remove SO_2 (alternate to N_2 purge)
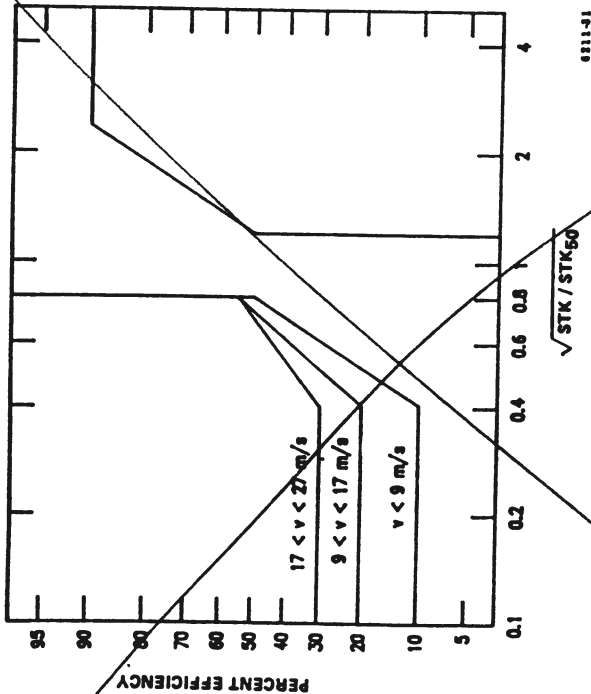
Figure 9. Efficiency envelope for first calibration stage.

METHOD 202—DETERMINATION OF CONDENSIBLE PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1. Applicability and Principle

1.1 Applicability. 1.1.1 This method applies to the determination of condensible particulate matter (CPM) emissions from stationary sources. It is intended to represent condensible matter as material that condenses after passing through a filter and as measured by this method (Note: The filter catch can be analyzed according to the appropriate method).

1.1.2 This method may be used in conjunction with Method 201 or 201A if the probes are glass-lined. Using Method 202 in conjunction with Method 201 or 201A, only the impinger train configuration and analysis is addressed by this method. The sample train operation and front end recovery and analysis shall be conducted according to Method 201 or 201A.

1.1.3 This method may also be modified to measure material that condenses at other temperatures by specifying the filter and probe temperature. A heated Method 5 out-of-stack filter may be used instead of the in-stack filter to determine condensible emissions at wet sources.

1.2 Principle. 1.2.1 The CPM is collected in the impinger portion of a Method 17 (appendix A, 40 CFR part 60) type sampling train. The impinger contents are immediately purged after the run with nitrogen (N_2) to remove dissolved sulfur dioxide (SO_2) gases from the impinger contents. The impinger solution is then extracted with methylene chloride ($MeCl_2$). The organic and aqueous fractions are then taken to dryness and the residues weighed. The total of both fractions represents the CPM.

1.2.2 The potential for low collection efficiency exist at oil-fired boilers. To improve the collection efficiency at these type of sources, an additional filter placed between the second and third impinger is recommended.

2. Precision and Interference

2.1 Precision. The precision based on method development tests at an oil-fired boiler and a catalytic cracker were 11.7 and 1.4 percent, respectively.

2.2 Interference. Ammonia. In sources that use ammonia injection as a control technique for hydrogen chloride (HCl), the ammonia interferes by reacting with HCl in the gas stream to form ammonium chloride (NH_4Cl) which would be measured as CPM. The sample may be analyzed for chloride and the equivalent amount of NH_4Cl can be subtracted from the CPM weight. However, if NH_4Cl is to be counted as CPM, the inorganic fraction should be taken to near dryness (less than 1 ml liquid) in the oven and

then allowed to air dry at ambient temperature to prevent any NH_4Cl from vaporizing.

3. Apparatus

3.1 Sampling Train. Same as in Method 17, section 2.1, with the following exceptions noted below (see Figure 202-1). Note: Mention of trade names or specific products does not constitute endorsement by EPA.

3.1.1 The probe extension shall be glass-lined or Teflon.

3.1.2 Both the first and second impingers shall be of the Greenburg-Smith design with the standard tip.

3.1.3 All sampling train glassware shall be cleaned prior to the test with soap and tap water, water, and rinsed using tap water, water, acetone, and finally, $MeCl_2$. It is important to completely remove all silicone grease from areas that will be exposed to the $MeCl_2$ during sample recovery.

3.2 Sample Recovery. Same as in Method 17, section 2.2, with the following additions:

3.2.1 N_2 Purge Line. Inert tubing and fittings capable of delivering 0 to 28 liters/min of N_2 gas to the impinger train from a standard gas cylinder (see Figure 202-2). Standard 0.95 cm (⅜-inch) plastic tubing and compression fittings in conjunction with an adjustable pressure regulator and needle valve may be used.

3.2.2 Rotameter. Capable of measuring gas flow at 20 liters/min.

3.3 Analysis. The following equipment is necessary in addition to that listed in Method 17, section 2.3:

3.3.1 Separatory Funnel. Glass, 1-liter.

3.3.2 Weighing Tins. 350-ml.

3.3.3 Dry Equipment. Hot plate and oven with temperature control.

3.3.4 Pipets. 5-ml.

3.3.5 Ion Chromatograph. Same as in Method 5F, Section 2.1.6.

4. Reagents

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. Where such specifications are not available, use the best available grade.

4.1 Sampling. Same as in Method 17, section 3.1, with the addition of deionized distilled water to conform to the American Society for Testing and Materials Specification D 1193-74, Type II and the omittance of section 3.1.4.

4.2 Sample Recovery. Same as in Method 17, section 3.2, with the following additions:

4.2.1 N_2 Gas. Zero N_2 gas at delivery pressures high enough to provide a flow of 20 liters/min for 1 hour through the sampling train.

4.2.2 Methylene Chloride. ACS grade. Blanks shall be run prior to use and only

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methylene chloride with low blank values (0.001 percent) shall be used.

4.2.3 Water. Same as in section 4.1.

4.3 Analysis. Same as in Method 17, section 3.3 with the following additions:

4.3.1 Methylene Chloride. Same as section 4.2.2.

4.3.2 Ammonium Hydroxide. Concentrated (14.8 M) NH₄OH.

4.3.3 Water. Same as in section 4.1.

4.3.4 Phenolphthalein. The pH indicator solution, 0.05 percent in 50 percent alcohol.

5. Procedure

5.1 Sampling. Same as in Method 17, section 4.1, with the following exceptions:

5.1.1 Place 100 ml of water in the first three impingers.

5.1.2 The use of silicone grease in train assembly is not recommended because it is very soluble in MeCl₂ which may result in sample contamination. Teflon tape or similar means may be used to provide leak-free connections between glassware.

5.2 Sample Recovery. Same as in Method 17, section 4.2 with the addition of a post-test N₂ purge and specific changes in handling of individual samples as described below.

5.2.1 Post-test N₂ Purge for Sources Emitting SO₂. (Note: This step is recommended, but is optional. With little or no SO₂ is present in the gas stream, i.e., the pH of the impinger solution is greater than 4.5, purging the impinger train is unnecessary.) As soon as possible after the post-test leak check, detach the probe and filter from the impinger train. Leave the ice in the impinger box to prevent removal of moisture during the purge. If necessary, add more ice during the purge to maintain the gas temperature below 20 °C. With no flow of gas through the clean purge line and fittings, attach it to the input of the impinger train (see Figure 202-2). To avoid over- or under-pressurizing the impinger array, slowly commence the N₂ gas flow through the line while simultaneously opening the meter box pump valve(s). When using the gas cylinder gas as the purge gas through the sample train, adjust the flow rate to 20 liters/min through the rotameter. When pulling the purge gas through the sample train using the meter box vacuum pump, set the orifice pressure differential to ΔH@ and maintain an overflow rate through the rotameter of less than 2 liters/min. This will guarantee that the N₂ delivery system is operating at greater than ambient pressure and prevents the possibility of passing ambient air (rather than N₂) through the impinger. Continue the purge under these conditions for 1 hour, checking the rotameter and ΔH value(s) periodically. After 1 hour, simultaneously turn off the delivery and pumping systems.

5.2.2 Sample Handling.

5.2.2.1 Container Nos. 1, 2, and 3. If filter catch is to be determined, as detailed in Method 17, section 4.2.

5.2.2.2 Container No. 4 (Impinger Contents). Measure the liquid in the first three impingers to within 1 ml using a clean graduated cylinder or by weighing it to within 0.5 g using a balance. Record the volume or weight of liquid present to be used to calculate the moisture content of the effluent gas. Quantitatively transfer this liquid into a clean sample bottle (glass or plastic); rinse each impinger and the connecting glassware, including probe extension, twice with water, recover the rinse water, and add it to the same sample bottle. Mark the liquid level on the bottle.

5.2.2.3 Container No. 5 (MeCl₂ Rinse). Follow the water rinses of each impinger and the connecting glassware, including the probe extension with two rinses of MeCl₂; save the rinse products in a clean, glass sample jar. Mark the liquid level on the jar.

5.2.2.4 Container No. 6 (Water Blank). Once during each field test, place 500 ml of water in a separate sample container.

5.2.2.5 Container No. 7 (MeCl₂ Blank). Once during each field test, place in a separate glass sample jar a volume of MeCl₂ approximately equivalent to the volume used to conduct the MeCl₂ rinse of the impingers.

5.3 Analysis. Record the data required on a sheet such as the one shown in Figure 202-3. Handle each sample container as follows:

5.3.1 Container Nos. 1, 2, and 3. If filter catch is analyzed, as detailed in Method 17, section 4.3.

5.3.2 Container Nos. 4 and 5. Note the level of liquid in the containers and confirm on the analytical data sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in Container No. 4 either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Remove a 5 ml aliquot and set aside for later ion chromatographic (IC) analysis of sulfates. (Note: Do not use this aliquot to determine chlorides since the HCl will be evaporated during the first drying step; Section 8.2 details a procedure for this analysis.)

5.3.2.1 Extraction. Separate the organic fraction of the sample by adding the contents of Container No. 4 (MeCl₂) to the contents of Container No. 4 in a 1000-ml separatory funnel. After mixing, allow the aqueous and organic phases to fully separate and drain off most of the organic/MeCl₂ phase. Then add 75 ml of MeCl₂ to the funnel, mix well, and drain off the lower organic phase. Repeat with another 75 ml of MeCl₂. This extraction should yield about 250 ml of organic extract. Each time, leave a

small amount of the organic/MeCl₂ phase in the separatory funnel ensuring that no water is collected in the organic phase. Place the organic extract in a tared 350-ml weighing tin.

5.3.2.2 Organic Fraction Weight Determination (Organic Phase from Container Nos. 4 and 5). Evaporate the organic extract at room temperature and pressure in a laboratory hood. Following evaporation, desiccate the organic fraction for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg.

5.3.2.3 Inorganic Fraction Weight Determination. (Note: If NH₄Cl is to be counted as CPM, the inorganic fraction should be taken to near dryness (less than 1 ml liquid) in the oven and then allow to air dry at ambient temperature. If multiple acid emissions are suspected, the ammonia titration procedure in section 8.1 may be preferred.) Using a hot plate, or equivalent, evaporate the aqueous phase to approximately 50 ml; then, evaporate to dryness in a 105 °C oven. Redissolve the residue in 100 ml of water. Add five drops of phenolphthalein to this solution; then, add concentrated (14.8 M) NH₄OH until the sample turns pink. Any excess NH₄OH will be evaporated during the drying step. Evaporate the sample to dryness in a 105 °C oven, desiccate the sample for 24 hours, weigh to a constant weight, and record the results to the nearest 0.1 mg. (Note: The addition of NH₄OH is recommended, but is optional when little or no SO₂ is present in the gas stream, i.e., when the pH of the impinger solution is greater than 4.5, the addition of NH₄OH is not necessary.)

5.3.2.4 Analysis of Sulfate by IC to Determine Ammonium Ion (NH₄⁺) Retained in the Sample. (Note: If NH₄OH is not added, omit this step.) Determine the amount of sulfate in the aliquot taken from Container No. 4 earlier as described in Method 5F (appendix A, 40 CFR part 60). Based on the IC SO₄⁻² analysis of the aliquot, calculate the correction factor to subtract the NH₄⁺ retained in the sample and to add the combined water removed by the acid-base reaction (see section 7.2).

5.3.3 Analysis of Water and MeCl₂ Blanks (Container Nos. 6 and 7). Analyze these sample blanks as described above in sections 5.3.2.3 and 5.3.2.2, respectively.

5.3.4 Analysis of Acetone Blank (Container No. 8). Same as in Method 17, section 4.3.

6. Calibration

Same as in Method 17, section 5, except for the following:

6.1 IC Calibration. Same as Method 5F, section 5.

6.2 Audit Procedure. Concurrently, analyze the audit sample and a set of compliance samples in the same manner to evaluate the technique of the analyst and the standards preparation. The same analyst, analytical reagents, and analytical system shall be used both for compliance samples and the EPA audit sample. If this condition is met, auditing of subsequent compliance analyses for the same enforcement agency within 30 days is not required. An audit sample set may not be used to validate different sets of compliance samples under the jurisdiction of different enforcement agencies, unless prior arrangements are made with both enforcement agencies.

6.3 Audit Samples. Audit Sample Availability. Audit samples will be supplied only to enforcement agencies for compliance tests. The availability of audit samples may be obtained by writing:

Source Test Audit Coordinator (MD-77B), Quality Assurance Division, Atmospheric Research and Exposure Assessment Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711

or by calling the Source Test Audit Coordinator (STAC) at (919) 541-7834. The request for the audit sample must be made at least 30 days prior to the scheduled compliance sample analysis.

6.4 Audit Results. Calculate the audit sample concentration according to the calculation procedure described in the audit instructions included with the audit sample. Fill in the audit sample concentration and the analyst's name on the audit response form included with the audit instructions. Send one copy to the EPA Regional Office or the appropriate enforcement agency and a second copy to the STAC. The EPA Regional Office or the appropriate enforcement agency will report the results of the audit to the laboratory being audited. Include this response with the results of the compliance samples in relevant reports to the EPA Regional Office or the appropriate enforcement agency.

7. Calculations

Same as in Method 17, section 6, with the following additions:

7.1 Nomenclature. Same as in Method 17, section 6.1 with the following additions.

C_{cw}=Concentration of the CPM in the stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).

C_{os}=Concentration of SO_4^{-2} in the sample, mg/ml.

m_a=Sum of the mass of the water and MeCl₂ blanks, mg.

m_b=Mass of the NH₄⁺ added to sample to form ammonium sulfate, mg.

m_1=Mass of inorganic CPM matter, mg.

m_o=Mass of organic CPM, mg.

m_s = Mass of dried sample from inorganic fraction, mg.
V_t = Volume of aliquot taken for IC analysis, ml.
V_s = Volume of impinger contents sample, ml.

7.2 Correction for NH_4^+ and H_2O. Calculate the correction factor to subtract the NH_4^+ retained in the sample based on the IC SO_4^{-2} and if desired, add the combined water removed by the acid-base reaction.

$$m_t = K \, C_{so2} \, V_{tc} \qquad \text{Eq. 202-1}$$

where:

K = 0.0205, when correcting for NH_4^+ and H_2O.
= 0.1840, when only correcting for NH_4^+.

7.3 Mass of Inorganic CPM.

$$m_i = m_r \frac{V_{tc}}{V_{tc} - V_b} - m_c \qquad \text{Eq. 202-2}$$

7.4 Concentration of CPM.

$$C_{cpm} = \frac{m_o + m_i - m_b}{VM_{std}} \qquad \text{Eq. 202-3}$$

8. Alternative Procedures

8.1 Determination of NH_4^+ Retained in Sample by Titration.

8.1.1 An alternative procedure to determine the amount of NH_4^+ added to the inorganic fraction by titration may be used. Begin by dissolving the inorganic residue in 100 ml of water, titrate the solution with 0.1 N NH_4OH to a pH of 7.0, as indicated by a pH meter. The 0.1 N NH_4OH is made as follows: Add 7 ml of concentrated (14.8 M) NH_4OH to 1 liter of water. Standardize against standardized 0.1 N H_2SO_4, and calculate the exact normality using a procedure parallel to that described in section 5.5 of Method 6 (appendix A, 40 CFR part 60). Alternatively, purchase 0.1 N NH_4OH that has been standardized against a National Institute of Standards and Technology reference material.

8.1.2 Calculate the concentration of SO_4^{-2} in the sample using the following equation.

$$CSO_4 = \frac{48.03 \, V_t N}{100} \qquad \text{Eq. 202-4}$$

where

N = Normality of the NH_4OH, mg/ml.

V_t = Volume of NH_4OH titrant, ml.
48.03 = mg/meq.
100 = Volume of solution, ml.

8.3.1 Calculate the CPM as described in section 7.

8.2 Analysis of Chlorides by IC. At the conclusion of the final weighing as described in section 5.3.2.3, redissolve the inorganic fraction in 100 ml of water. Analyze an aliquot of the redissolved sample for chlorides by IC using techniques similar to those described in Method 5F for sulfates. Previous drying of the sample should have removed all HCl. Therefore, the remaining chlorides measured by IC can be assumed to be NH_4Cl, and this weight can be subtracted from the weight determined for CPM.

8.3 Air Purge to Remove SO_2 from Impinger Contents. As an alternative to the post-test N_2 purge described in section 5.2.1, the tester may opt to conduct the post-test purge with air at 20 liter/min. Note: The use of an air purge is not as effective as a N_2 purge.

8.4 Chloroform-ether Extraction. As an alternative to the methylene chloride extraction described in section 5.3.2.1, the tester may opt to conduct a chloroform-ether extraction. Note: The Chloroform-ether extraction was not as effective as the $MeCl_2$ in removing the organics, but it was found to be an acceptable organic extractant. Chloroform and diethylether of ACS grade, with low blank values (0.001 percent), shall be used. Analysis of the chloroform and diethylether blanks shall be conducted according to Section 5.3.3 for $MeCl_2$.

8.4.1 Add the contents of Container No. 4 to a 1000-ml separatory funnel. Then add 75 ml of chloroform to the funnel, mix well, and drain off the lower organic phase. Repeat two more times with 75 ml of chloroform. Then perform three extractions with 75 ml of diethylether. This extraction should yield approximately 450 ml of organic extraction. Each time, leave a small amount of the organic/$MeCl_2$ phase in the separatory funnel ensuring that no water is collected in the organic phase.

8.4.2 Add the contents of Container No. 5 to the organic extraction. Place approximately 300 ml of the organic extract in a tared 350-ml weighing tin while storing the remaining organic extract in a sample container. As the organic extract evaporates, add the remaining extract to the weighing tin.

8.4.3 Determine the weight of the organic phase as described in Section 5.3.2.2.

8.5 Improving Collection Efficiency. If low impinger collection efficiency is suspected, the following procedure may be used.

8.5.1 Place an out-of-stock filter as described in Method 8 between the second and third impingers.

8.5.2 Recover and analyze the filter according to Method 17, Section 4.2. Include the filter holder as part of the connecting glassware and handle as described in sections 5.2.2.2 and 5.2.2.3.

8.5.3 Calculate the Concentration of CPM as follows:

$$C_{cpm} = \frac{m_o + m_i + m_f - m_b}{VM_{std}} \qquad \text{Eq. 202-5}$$

where:

m_f = amount of CPM collected on out-of-stack filter, mg.

8.6 Wet Source Testing. When testing at a wet source, use a heated out-of-stack filter as described in Method 5.

9. Bibliography

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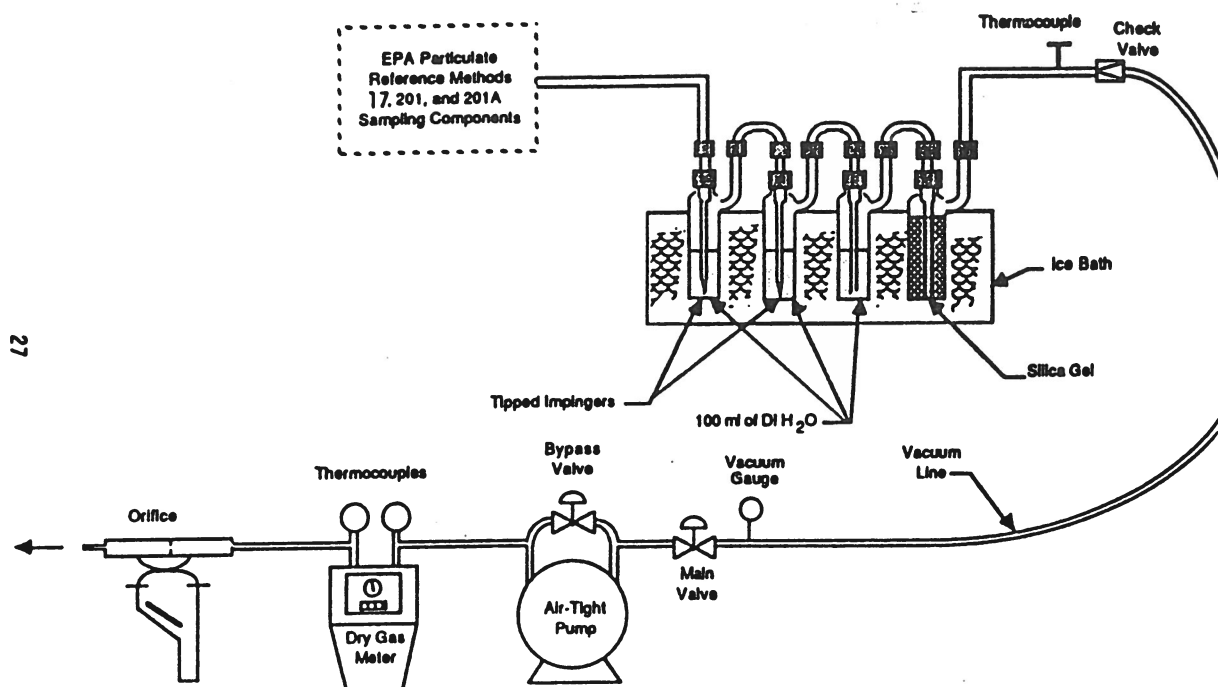
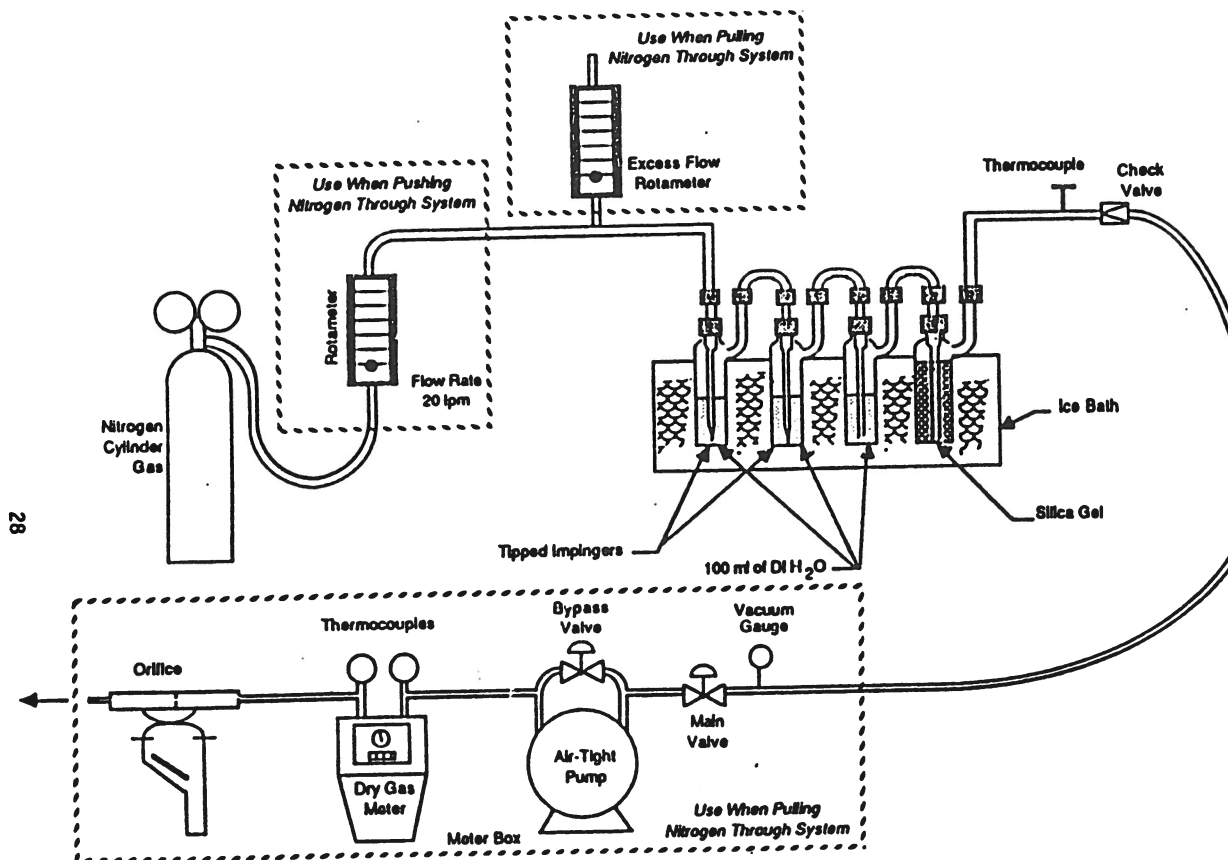Figure 202-1. Schematic of condensible particulate sampling train.

Figure 202-2. Schematic of post-test nitrogen purge system.